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by

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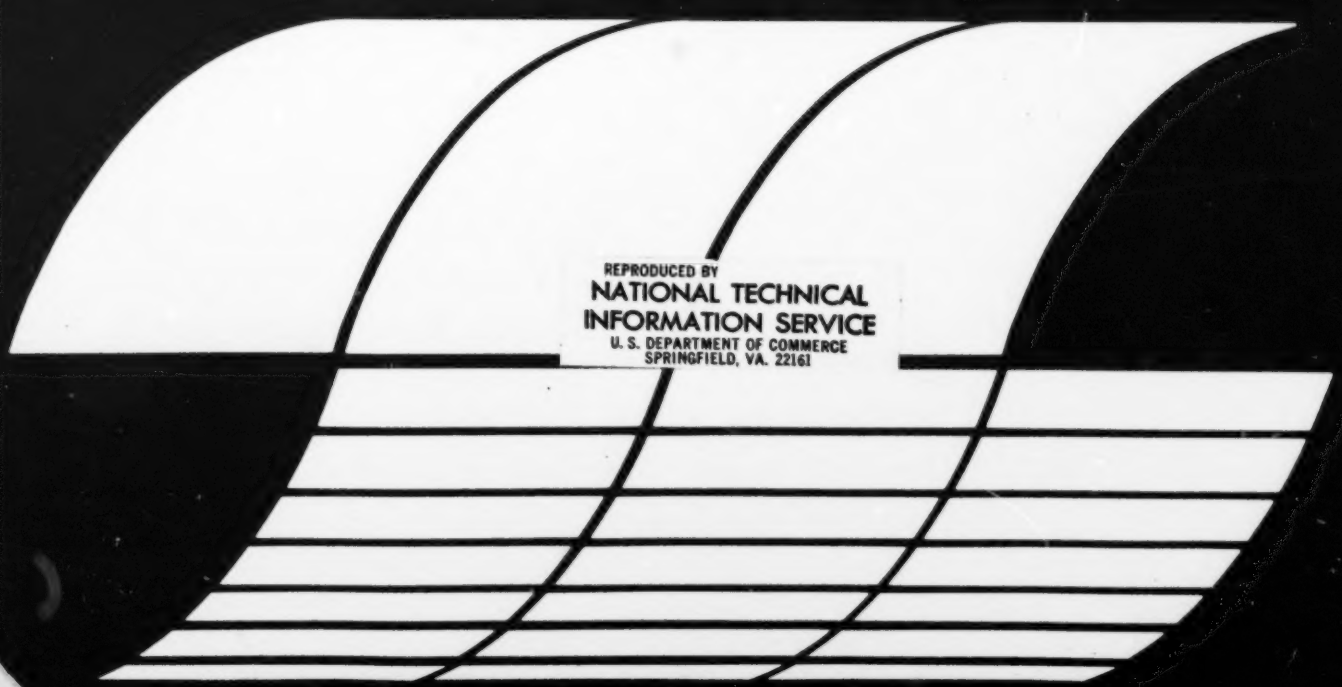

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ABSTRACT

This report presents the results of a washability study showing the trace element contents of various specific gravity fractions for 10 coal samples collected from various coal-producing regions of the United States.

Reliable analytical methods were developed to determine cadmium, chromium, copper, fluorine, mercury, manganese, nickel, and lead in the whole coals and the various specific gravity fractions of the coals.

The material balances for the 8 trace elements for the 10 coals ranged from 85 to 115 percent with an average of 99 percent and a 95-percent confidence interval of ±3 percent.

The magnitude of the concentrations of the various trace elements varied quite a bit from coalbed to coalbed within a region and also from region to region.

The data from the analytical determinations on the washed coals are plotted as washability curves so that the quantity and quality of the clean coal products can be obtained at the desired specific gravity of separation.

Generally, the data showed that most of the trace elements of interest concentrated in the heavier specific gravity fractions of the coal, indicating that they are associated with mineral matter; removal of this material should result in significant trace element reductions, ranging up to 88 percent.

A list of references of other studies of trace elements in coal is presented.

CONTENTS

	Page
Abstract	iii
Figures	v
Tables	vi
Acknowledgments	viii
Introduction	1
Experimental Work	1
Sample Collection	1
Sample Preparation	1
Gravimetric Testing	2
Analytical Methods	2
Methods Employed	4
Experimental Results: Washability Data	6
Discussion of Results	14
Northern Appalachian Region Coals	14
Southern Appalachian Region Coals	18
Eastern Midwest Region Coals	18
Western Region Coals	23
Conclusions	23
References	28

FIGURES

Number		Page
1	Washability analyses of Pittsburgh bed coal, Allegheny County, Pa., showing the trace element content at various specific gravities of separation and clean coal recoveries	15
2	Washability analyses of Waynesburg bed coal, Belmont County, Ohio, showing the trace element content at various specific gravities of separation and clean coal recoveries	16
3	Washability analyses of Upper Freeport bed coal, Garrett County, Md., showing the trace element content at various specific gravities of separation and clean coal recoveries	17
4	Washability analyses of Hazard No. 4 bed coal, Bell County, Ky. (East), showing the trace element content at various specific gravities of separation and clean coal recoveries	19
5	Washability analyses of No. 6 bed coal, Perry County, Ill., showing the trace element content at various specific gravities of separation and clean coal recoveries	20
6	Washability analyses of No. 5 bed coal, Perry County, Ill., showing the trace element content at various specific gravities of separation and clean coal recoveries	21
7	Washability analyses of No. 7 bed coal, Ohio County, Ky. (West), showing the trace element content at various specific gravities of separation and clean coal recoveries	22
8	Washability analyses of Red bed coal, Navajo County, Ariz., showing the trace element content at various specific gravities of separation and clean coal recoveries	24
9	Washability analyses of No. 8 bed coal, San Juan County, N. Mex., showing the trace element content at various specific gravities of separation and clean coal recoveries	25
10	Washability analyses of Rock Springs No. 3 bed coal, Sweetwater County, Wyo., showing the trace element content at various specific gravities of separation and clean coal recoveries	26

TABLES

Number		Page
1	Analysis of NBS standard reference coals, 1630 and 1632	3
2	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Pittsburgh coalbed, Pennsylvania	7
3	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Waynesburg coalbed, Ohio . . .	7
4	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Upper Freeport coalbed, Maryland	8
5	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Hazard No. 4 coalbed, Kentucky (East)	8
6	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, No. 6 coalbed, Illinois . . .	9
7	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, No. 5 coalbed, Illinois . . .	9
8	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, No. 7 coalbed, Kentucky (West)	10
9	Washability analyses showing the levels of trace elements in the sample crushed to 3/8-inch top size, Red coalbed, Arizona	10
10	Washability analyses showing the levels of trace elements in the sample crushed to 3/8-inch top size, No. 8 coalbed, New Mexico . .	11
11	Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Rock Springs No. 3 coalbed, Wyoming	11

Number Page

12 Summary of composite product analyses by region for coals crushed to 14-mesh top size and cleaned at 1.60 specific gravity, showing the trace element reduction attainable 12

13 Summary of product analyses showing the ratio of trace element concentration of the float 1.60 specific gravity product to that of the sink 1.60 specific gravity product 13

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INTRODUCTION

There is a general awareness that trace elements in coal might contribute substantial quantities of potentially hazardous materials to the environment. Most of the 650 million tons of coal mined goes to powerplants where it is burned. Thus, a coal containing concentrations of only 1 part per million (ppm) could emit 650 tons per year of a potentially hazardous substance into the environment.

Certain trace elements may concentrate in particular specific gravity fractions of the raw coal; those that do, may be removed by conventional coal-washing processes prior to combustion.

This report discusses (1) the development of reliable analytical methods for quantifying eight trace elements in raw coals and in their various specific gravity fractions (During informal discussions with the Environmental Protection Agency, they provided a list of 17 potentially toxic elements in coal; this report evaluates 8 of those elements, Cd, Cr, Cu, F, Hg, Mn, Ni, and Pb, and work is continuing on the remaining 9 elements), (2) the results of washability analyses performed on selected coals to show the distribution of the trace elements in the various specific gravity fractions, and (3) the evaluation of the data to determine if the trace element concentrations of the coals could be reduced by removal of selected specific gravity increments.

EXPERIMENTAL WORK

Sample Collection

Face samples were collected according to the procedure recommended by Fieldner and Selvig (10) and Holmes (12), except that the dimensions of each sample cut were expanded to permit 600 pounds of coal to be taken from the face. Partings and impurities were not removed from the samples. The samples were loaded into drums which contained plastic liners and shipped to the Bureau of Mines coal preparation laboratories at Bruceton, Pa., for analysis.

Sample Preparation

Each 600-pound channel sample was air-dried and then crushed to 1-1/2-inch top size. The sample was then coned, long-piled, shoveled into four pans, and divided into two portions by combining opposite pans. One of the portions was crushed and riffled in several stages until a 3-pound sample of 14-mesh by 0 material was obtained for washability analysis. This procedure was followed for all samples except those from Arizona and New Mexico. For these samples the material crushed to 3/8-inch top size was used since the 14-mesh by 0 material had been inadvertently discarded. The sample was then float-sink

tested in glass separatory vessels at 1.30, 1.40, and 1.60 specific gravities using CERTIGRAV, a commercial organic liquid; the solution tolerance is ± 0.001 specific gravity unit and was monitored using a spindle hydrometer.

Gravimetric Testing

The sample was placed in the 1.30 specific gravity bath in small quantities to prevent particle entrapment, stirred, and allowed to separate. The lighter density coal fraction was removed from the surface of the bath by vacuum filtration, and the heavier density material which settled to the container bottom was also vacuum-filtered. The heavier density material was then placed in the next higher specific gravity solution, and the process was repeated until the sample was separated into the desired specific gravity fractions.

Upon completion of the float-sink testing, the specific gravity fractions were air-dried and analyzed for cadmium, chromium, copper, fluorine, mercury, manganese, nickel, and lead. All results are the average of at least two chemical analyses.

It should be noted that float-sink separations are based on the specific gravity of the heterogeneous particles separated. If individual components of the coal are small enough in size and are physically attached to larger particles, they will be separated into a specific gravity fraction that is the average of the weighted specific gravity of the two particles. The finer a coal is ground, the greater the liberation of the individual constituents having different specific gravities, and the sharper the separation of these particles. In coal preparation practice today, most coals are not crushed finer than about 1-inch top size. Since the tests conducted in this study were designed to simulate current practice, some of the particles float-sink tested were not discrete particles of pyrite, rock, or coal. These heterogeneous particles lead to what may be interpreted as anomalous results. Although this effect cannot be wholly eliminated, replicate analyses, which were performed in this study, can help define the trace element content of the various specific gravity fractions.

The float-sink data from the channel samples are not to be construed as representing the quality of the product loaded at the mine where the sample was taken but rather as indicating the quality of the bed in that particular geographical location. Float-sink data are based upon theoretically perfect specific gravity separations, which are approached but not equaled in commercial practice.

Analytical Methods

The complex and variable nature of coal makes any reliable chemical analysis difficult. Add to the normal difficulties of coal analysis the many pitfalls of trace element analysis, and the analyst must exercise extreme care in order to produce precise and accurate results. The chemical analyses in the present study were performed using standard coals, material balances, and the method of standard additions in an effort to produce results that could be presented with some degree of confidence.

Contamination: A major concern for an analyst engaged in trace element analyses is contamination. Additions of extremely small amounts of extraneous material to a sample may yield erroneous results. The contamination of samples may occur during storage, handling, or analysis.

Another source of contamination was found to be automobile exhaust products in airborne dust. Lead from these exhaust products will be deposited on samples left out in the laboratory, particularly if the laboratory is located near heavily traveled roads.

Mercury is ubiquitous in most laboratories and will contaminate samples or equipment left out in the laboratory for any length of time. Mercury vapor is also present in tanks of laboratory gases but can be eliminated by the use of a charcoal filter in lines carrying the gases.

Many contamination problems can be eliminated by proper precautions if one is aware of their presence. Others must be accounted for in blank corrections with a resultant loss of precision.

Losses: Another source of error in trace element analysis is loss of the analyte through contact with an adsorbing surface. An interesting example of this "negative contamination" was observed with the use of "nonwetting" platinum crucibles that were tested for use in the lithium metaborate fusion procedure described below. The use of these crucibles, which are fabricated of a platinum-5 percent gold alloy, results in serious loss of trace copper. The results of a series of tests with these crucibles showed that the concentration of copper in the lithium metaborate was reduced from 16 ± 3 μg Cu/g to 4 ± 1 μg Cu/g when the lithium metaborate was fused in the "nonwetting" crucibles. No loss was observed when the lithium metaborate was fused in standard platinum crucibles.

Analysis of Standard Coals: The National Bureau of Standards has certified two coals for trace element content. SRM 1630 is certified for mercury, and SRM 1632 is certified for 14 trace elements. The trace element concentrations for the standard coals as determined in this laboratory are shown in table 1.

TABLE 1. - Analysis of NBS Standard Reference Coals, 1630 and 1632

SRM No.	Element	Certified value (ppm)[1]	Determined value (ppm)[2]
1630	Mercury	0.126 ± 0.006	0.125 ± 0.014
1632	Mercury	0.12 ± 0.02	0.10 ± 0.01
1632	Cadium	0.19 ± 0.03	0.17 ± 0.02
1632	Lead	30 ± 9	28 ± 3
1632	Nickel	15 ± 1	15 ± 2
1632	Copper	18 ± 2	16.7 ± 0.6
1632	Chromium	20.2 ± 0.5	20.1 ± 0.6
1632	Manganese	40 ± 3	45.8 ± 0.8

[1]Statistic defined by NBS as " ... in no case less than the 95 percent confidence limits computed for the analyses."
[2]1 standard deviation.

Neither of the NBS standard coals was certified for fluorine so SRM 56b, a phosphate rock containing 3.4 percent fluorine, was used as the standard. The fluorine content was found to be 3.3 percent ± 0.1 percent.

Material Balances: The study of float-sink fractions affords an additional check on the analytical methods; namely, material balances. For each element studied, the sum of the trace element content found in the various specific gravity fractions should agree with the trace element content found in the starting coal. Having no objective guidelines at the start of this work, a rejection criterion of ±15 percent was arbitrarily set on the material balance for each element. Thus, if a material balance did not fall between 85 percent and 115 percent for an element in a coal, the analyses were repeated.

After processing all 10 coals, the average material balance was calculated to be 99 percent with a 95-percent confidence interval of ±3 percent. The average was calculated by including all material balances regardless of their value, provided no objective reason was known for their rejection. An example of an objective reason for rejection would be known contamination of a sample.

Method of Standard Additions: As mentioned earlier, the analysis of coals is difficult owing to their nonuniformity. The nonuniformity of samples is even greater in the float-sink fractions, where each fraction is chemically quite different from all the others. No single standard material can be devised to represent the varying chemical matrix in a series of float-sink samples. To overcome this difficulty, the method of standard additions was employed in all the analyses except for the determination of mercury and fluorine, where isolation of the analyte element from the sample matrix is part of the experimental method.

The method of standard additions overcomes matrix effects by utilizing the sample itself as the standard. This is accomplished by splitting the sample solution into four equal parts. Known amounts of the element of interest are added to three of the sample aliquots. The additions are contained in volumes that are small compared with the total volume of the sample solution, so that dilution of the sample solution is negligible. When the four solutions are analyzed, the absorption is plotted as the ordinate and the amount of analyte added to each solution is plotted as the abscissa. The result should be a straight line with a negative intercept. The magnitude of the intercept is equal to the amount of analyte present in the original sample solution. In practice, the plotting is accomplished on a programmable desk calculator using a linear regression by the method of least squares.

Methods Employed

As the details of the analytical methods employed in this study will be published separately, the descriptions to follow will only outline the procedures.

Mercury: Mercury was determined by a double gold amalgamation-atomic absorption procedure that has been described previously (9, 19).

During this study it was discovered that several of the sink 1.60 samples derived from high-sulfur coals produced H_2SO_4 when combusted in oxygen. The sulfuric acid tended to coat the gold wire used in the amalgamators, resulting in loss of sensitivity. To overcome this difficulty, a modified procedure was adopted in which a nitrogen pyrolysis method was used to analyze samples that when burned in oxygen caused a loss of sensitivity due to the formation of H_2SO_4. The sample was pyrolyzed in a nitrogen stream, and the resultant gases were burned in an oxygen atmosphere. After oxidation, the gases were processed in the same manner as the gases that were produced by burning coal in an oxygen stream.

Fluorine: A number of methods are reported in the literature for the determination of fluorine in coal (2, 5, 8, 15). Most of them involve the bleaching action of fluoride ion on a colored complex. The methods are subject to numerous interferences, and generally the colored complexes are not stable for appreciable amounts of time. The method employed in this study utilizes a fluoride-ion-specific electrode, which is simpler and faster to use and is not as subject to interferences as were the methods used in the past.

A 2-gram coal sample was mixed with 0.8 gram of CaO in a platinum crucible and ashed at 600° C until all carbonaceous matter was oxidized. The residue was fused with 4 grams of Na_2CO_3. The fusion cake was leached with phosphoric acid, and the fluorine was distilled from a phosphoric acid-sulfuric acid mixture at 135° C. The distillate was made basic to phenolphthalein with 1 percent sodium carbonate solution and evaporated to about 5 ml. The solution was neutralized with 1:1 H_2SO_4 using methyl orange as an indicator. Ten milliliters of a commercial fluoride-ion-electrode buffer was added, and the volume was adjusted to 25 ml with distilled deionized water. The solution was transferred to a plastic beaker, and the potential measurements were made on an expanded-scale pH meter.

At fluoride concentrations below 10^{-4} molar, the electrode response does not follow the Nernst relationship (that is, the electrode response is not linearly related to the logarithm of the fluoride concentration), and it becomes necessary to add a known quantity of fluoride to the solution in order to bring the concentration into the linear range of electrode response. After the concentration of fluoride in the solution has been determined, the fluoride addition that has been made is subtracted; the difference is the fluoride ion concentration in the solution from the sample.

Cadmium and Lead: Cadmium is normally present in coal at concentrations well below 1 ppm, and while lead concentrations can run much higher, the atomic absorption sensitivity for an aqueous solution of lead is only about 0.5 µg/ml for 1 percent absorption. Solvent extraction offers a means for increasing the sensitivity of the determinations by isolation and concentration of the metals of interest. A procedure was developed utilizing the extraction of the iodide complexes of lead and cadmium into methylisobutylketone (MIBK) (13).

A 10-gram sample of coal (or a 5-gram sample of the sink 1.60 fraction) was weighed into a Vycor or platinum dish and placed in a cold muffle furnace. The temperature was raised to 500° C in 1 hour, and ashing continued at that temperature for about 16 hours. Tests in this and other laboratories (3, 17) showed no significant loss at this temperature for the trace metals investigated. The resulting ash was digested in concentrated HCl and filtered, and

the insoluble residue was again ashed at 500° C. After treatment with HF and H_2SO_4 to volatilize silica, the sample was again leached with HCl and filtered. The residue after ignition was fused in potassium carbonate, the fusion cake was dissolved in dilute HCl, the resulting solution was added to the combined filtrates, and the solution was evaporated to near dryness. The residue was dissolved in HCl, transferred to a volumetric flask, and diluted to volume. Aliquots were taken, and standard additions of lead and cadmium were made. Ascorbic acid, potassium iodide, and MIBK were added, and the lead and cadmium iodides were extracted into the MIBK. The ketone layer was aspirated into the flame of an atomic absorption spectrophotometer. Methyl isobutyl ketone was used to establish a base line. The amount of analyte present was calculated by means of a linear least squares regression procedure.

Chromium, Copper, Manganese, and Nickel: The procedure used for the preparation of coal samples for the determination of Cr, Cu, Mn, and Ni was similar to that used for the preparation of coal samples for Cd and Pb determinations. A 2-gram sample of coal (or a 1-gram sample of the sink 1.60 fraction) was ashed at 500° C in a platinum crucible, and the ash was then treated with H_2SO_4 and HF to volatilize the silica. After evaporation, the residue was leached with concentrated HCl and filtered. If considerable residue remained, it was again treated with HF and leached with HCl. Finally the insoluble portion was fused at 950° C with $LiBO_2$. The fusion cake, after cooling, was dissolved in 3 N HCl, and the solution was combined with the filtrates. The solution was transferred to a 100 ml volumetric flask and diluted to volume. Three 25 ml aliquots were taken, and additions of standards were made to each aliquot. As was done with the lead and cadmium determinations, a linear least squares regression was used to calculate the concentration of analyte present.

EXPERIMENTAL RESULTS: WASHABILITY DATA

Ten sets of individual washability data were compiled to show the fate of the various trace elements in the coals tested upon crushing to 14-mesh top size and subsequent specific gravity fractionation (tables 2 through 11). Three samples each were evaluated from the Northern Appalachian Region, the Eastern Midwest Region, and the Western Region; one sample from the Southern Appalachian Region was also evaluated.

Each set of washability data shows the direct and cumulative weight-percents of each specific gravity fraction and the trace element contents of each fraction in parts per million. The trace element contents of the head sample are also shown for comparative purposes with the composite washability analyses.

Generally, the magnitude of the various trace element content levels, as determined in the whole coals from the various regions, were comparable to those as determined by R. R. Ruch et al. (18). A summary of composite product analyses by region is presented in table 12.

Table 13 is a summary of the product analyses expressing the trace element content as a ratio of the trace element concentration of the float 1.60 specific gravity product to the trace element concentration of the sink 1.60 specific gravity product:

TABLE 2. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Pittsburgh coalbed, Pennsylvania

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	59.4	0.03	11	4.6	17	0.08	2.8	7.4	1.7	59.4	0.03	11	4.6	17	0.08	2.8	7.4	1.7
1.30 -1.40	29.3	.09	19	6.7	33	.09	5.9	10	3.9	88.7	.05	14	5.2	22	.08	3.8	8.2	2.4
1.40 -1.60	5.9	.35	31	19	81	.28	19	15	13	94.6	.07	15	6.1	26	.10	4.8	8.7	3.1
Sink -1.60	5.4	.39	43	43	125	1.7	150	30	26	100.0	.09	16	8.1	31	.18	13	9.8	4.3
Head sample	-	-	-	-	-	-	-	-	-	100.0	.09	16	9.0	35	.19	11	11	4.3

Cd = cadmium, Cr = chromium, Cu = copper, F = fluorine, Hg = mercury, Mn = manganese, Ni = nickel, and Pb = lead.

TABLE 3. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Waynesburg coalbed, Ohio

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	23.4	0.14	15	6.1	27	0.13	4.3	8.1	2.1	23.4	0.14	15	6.1	27	0.13	4.3	8.1	2.1
1.30 -1.40	40.7	.06	18	5.6	53	.07	8.2	9.6	2.4	64.1	.09	17	5.8	44	.09	6.8	9.0	2.3
1.40 -1.60	20.6	.15	24	10	113	.15	20	12	5.6	84.7	.10	19	6.8	60	.10	10	9.8	3.1
Sink -1.60	15.3	.36	30	41	146	.61	66	41	26	100.0	.14	20	12	73	.18	19	15	6.6
Head sample	-	-	-	-	-	-	-	-	-	100.0	.14	21	11	78	.18	18	16	6.7

TABLE 4. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Upper Freeport coalbed, Maryland

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	37.6	0.07	13	7.0	8	0.08	2.5	8.1	0.8	37.6	0.07	13	7.0	8	0.08	2.5	8.1	0.8
1.30 -1.40	36.7	.06	23	8.8	43	.16	6.5	9.2	2.6	74.3	.06	18	7.9	25	.12	4.5	8.6	1.7
1.40 -1.60	10.3	.20	34	24	80	.56	23	26	9.2	84.6	.08	20	9.8	32	.17	6.7	11	2.6
Sink -1.60	15.4	.25	73	58	[1]279	1.13	51	38	36	100.0	.10	28	17	[1]70	.32	14	15	7.7
Head sample	-	-	-	-	-	-	-	-	-	100.0	.10	27	16	70	.28	13	16	6.5

[1]Insufficient sample; however, this number was calculated by compositing the other fractions and making a material balance.

TABLE 5. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Hazard No. 4 coalbed, Kentucky (East)

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	51.0	0.08	6	13	11	0.04	30	10	3.5	51.0	0.08	6	13	11	0.04	30	10	3.5
1.30 -1.40	16.9	.20	11	26	26	.07	89	15	9.7	67.9	.11	7	16	15	.05	45	11	5.0
1.40 -1.60	9.2	.24	33	55	110	.12	240	28	25	77.1	.12	10	21	26	.06	68	13	7.4
Sink -1.60	22.9	.10	73	66	400	.22	1,100	38	40	100.0	.12	25	31	112	.09	300	19	15
Head sample	-	-	-	-	-	-	-	-	-	100.0	.12	26	28	110	.09	260	18	14

TABLE 6. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, No. 6 coalbed, Illinois

Product	Direct: Weight, percent	Direct: Parts per million Cd	Cr	Cu	F	Hg	Mn	Ni	Pb	Cumulative: Weight, percent	Cumulative: Parts per million Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	44.1	0.00	10	2.7	40	0.06	8.4	11	2.4	44.1	0.00	10	2.7	40	0.06	8.4	11	2.4
1.30 -1.40	29.5	.01	17	6.4	75	.08	14	18	8.2	73.6	.00	13	4.2	52	.07	11	13	4.7
1.40 -1.60	13.9	.09	19	9.4	120	.12	22	22	13	87.5	.02	14	5.0	65	.08	12	15	6.0
Sink -1.60	12.5	5.00	29	26	150	.15	230	26	40	100.0	.64	16	7.6	76	.09	39	16	10
Head sample	-	-	-	-	-	-	-	-	-	100.0	.61	16	8.6	79	.09	37	18	9.5

TABLE 7. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, No. 5 coalbed, Illinois

Product	Direct: Weight, percent	Direct: Parts per million Cd	Cr	Cu	F	Hg	Mn	Ni	Pb	Cumulative: Weight, percent	Cumulative: Parts per million Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	49.3	0.02	8.5	4.6	18	0.06	9	7.4	2.3	49.3	0.02	8.5	4.6	18	0.06	9	7.4	2.3
1.30 -1.40	31.8	.06	14	7.0	39	.04	15	6.9	3.4	81.1	.04	11	5.5	26	.06	11	7.2	2.7
1.40 -1.60	12.1	.16	13	11	72	.04	41	10	3.7	93.2	.05	11	6.2	32	.05	15	7.6	2.8
Sink -1.60	6.8	4.3	6.0	18	63	.09	1,100	1.1	6.0	100.0	.34	11	7.0	34	.06	88	7.1	3.1
Head sample	-	-	-	-	-	-	-	-	-	100.0	.33	12	6.3	37	.06	89	8.2	3.0

TABLE 8. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, No. 7 coalbed, Kentucky (West)

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	38.7	0.01	5.8	1.6	32	0.06	6.2	4.9	1.0	38.7	0.01	5.8	1.6	32	0.06	6.2	4.9	1.0
1.30 -1.40	39.8	.03	8.8	4.4	48	.09	11	6.6	2.7	78.5	.02	7.3	3.0	40	.07	11	5.8	1.9
1.40 -1.60	15.1	.06	13	7.9	91	.19	21	9.2	5.1	93.6	.03	8.2	3.8	48	.09	11	6.3	2.4
Sink -1.60	6.4	.29	20	13	100	.55	47	7.6	17	100.0	.04	9.0	4.4	52	.12	13	6.4	3.3
Head sample	-	-	-	-	-	-	-	-	-	100.0	.06	9.6	5.0	57	.13	15	6.6	3.7

TABLE 9. - Washability analyses showing the levels of trace elements in the sample crushed to 3/8-inch top size, Red coalbed, Arizona

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	53.6	0.05	2.2	2.0	11	0.03	13	3.1	2.7	53.6	0.05	2.2	2.0	11	0.03	13	3.1	2.7
1.30 -1.40	35.6	.07	4.2	4.9	21	.04	14	2.9	5.5	89.2	.06	3.0	3.2	15	.03	13	3.0	3.8
1.40 -1.60	6.6	.07	10	13	41	.03	22	3.3	14	95.8	.06	3.5	3.8	17	.03	14	3.0	4.5
Sink -1.60	4.2	.18	10	15	86	.08	26	4.0	21	100.0	.06	3.8	4.3	20	.04	14	3.1	5.2
Head sample	-	-	-	-	-	-	-	-	-	100.0	.07	3.7	4.8	21	.04	14	3.0	5.8

TABLE 10. - Washability analyses showing the levels of trace elements in the sample crushed to 3/8-inch top size, No. 8 coalbed, New Mexico

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	31.2	0.03	3.8	8.2	46	0.03	15	3.2	3.4	31.2	0.03	3.8	8.2	46	0.03	15	3.2	3.4
1.30 -1.40	31.2	.03	4.9	13	35	.04	18	3.8	7.8	62.4	.03	4.4	11	40	.04	16	3.5	5.6
1.40 -1.60	19.6	.09	6.7	16	40	.05	37	3.3	13	82.0	.04	4.9	12	40	.04	21	3.4	7.4
Sink -1.60	18.0	.27	3.2	25	110	.18	330	2.7	26	100.0	.08	4.6	14	53	.06	77	3.3	11
Head sample	-	-	-	-	-	-	-	-	-	100.0	.08	5.0	13	52	.07	88	3.4	12

TABLE 11. - Washability analyses showing the levels of trace elements in the sample crushed to 14-mesh top size, Rock Springs No. 3 coalbed, Wyoming

Product	Direct									Cumulative								
	Weight, percent	Parts per million								Weight, percent	Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Float-1.30	60.7	0.06	0.85	3.6	33	0.05	14	3.9	1.9	60.7	0.06	0.85	3.6	33	0.05	14	3.9	1.9
1.30 -1.40	24.1	.13	3.7	4.5	46	.08	14	5.9	4.1	84.8	.08	1.7	3.9	37	.06	14	4.5	2.5
1.40 -1.60	9.6	.61	8.3	9.4	64	.06	18	15	10	94.4	.13	2.3	4.4	39	.06	14	5.5	3.3
Sink -1.60	5.6	.61	8.5	7.9	180	.19	540	13	34	100.0	.16	2.7	4.6	47	.07	44	6.0	5.0
Head sample	-	-	-	-	-	-	-	-	-	100.0	.15	3.1	4.3	51	.07	47	5.2	4.7

TABLE 12. - Summary of composite product analyses by region for coals crushed to 14-mesh top size and cleaned at 1.60 specific gravity, showing the trace element reduction attainable

Product	Yield, percent	Cumulative analyses, Parts per million							
		Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Northern Appalachian Region									
Float 1.60	88	0.08	18	8	39	0.12	7	10	2.9
Composite washability	100	.11	21	12	58	.23	12	13	6.2
Reduction, percent	-	27	14	33	33	48	42	23	53
Southern Appalachian Region									
Float 1.60	77	0.12	10	21	26	0.06	68	13	8
Composite washability	100	.12	25	31	110	.09	300	19	15
Reduction, percent	-	0	60	32	77	33	77	32	47
Eastern Midwest Region									
Float 1.60	91	0.03	11	5	48	0.07	13	9.6	3.7
Composite washability	100	.34	12	6.3	54	.09	47	9.8	5.6
Reduction, percent	-	88	8	21	11	22	72	2	34
Western Region									
Float 1.60	91	0.07	3.6	6.7	32	0.04	16	4.0	5.1
Composite washability	100	.10	3.7	7.6	40	.06	45	4.1	7.1
Reduction, percent	-	30	3	12	20	33	64	2	28

TABLE 13. - Summary of product analyses showing the ratio[1] of trace element concentration of the float 1.60 specific gravity product to that of the sink 1.60 specific gravity product

Product	Ratios							
	Cd	Cr	Cu	F	Hg	Mn	Ni	Pb
Northern Appalachian Region								
Float 1.60--	1/4	1/3	1/6	1/5	1/10	1/13	1/4	1/10
Sink 1.60--								
Southern Appalachian Region								
Float 1.60--	1/1	1/7	1/3	1/15	1/4	1/16	1/3	1/5
Sink 1.60--								
Eastern Midwest Region								
Float 1.60--	1/320	1/2	1/4	1/2	1/4	1/35	1/1	1/6
Sink 1.60--								
Western Region								
Float 1.60--	1/5	1/2	1/2	1/4	1/4	1/19	1/2	1/5
Sink 1.60--								

[1]It should be noted that the ratio does not reflect weight balances but only trace element concentrations in each fraction.

(trace element concentration of float 1.60 specific gravity product)
(trace element concentration of sink 1.60 specific gravity product)

These are interesting numbers because the samples tested were raw coal channel samples and therefore did not include any roof or floor material. The yields of sink 1.60 specific gravity product (refuse material) for the four regions tested averaged 12, 23, 9, and 9 percent. Under normal mining conditions, on the average, 25 percent of the mined raw coal will report to the sink 1.60 specific gravity product. Thus using this criterion and assuming that the trace element content of the roof and floor material would be in the same concentration as in the sink 1.60 specific gravity material in the raw coal channel sample, it can be seen that the percent of trace element reduction would be greater than that shown for the coals tested in the four regions.

DISCUSSION OF RESULTS

Northern Appalachian Region Coals

Three coalbed samples collected from Pennsylvania (1), Ohio (1), and Maryland (1) were evaluated; the washability data are presented in tables 2 through 4 and plotted in figures 1 through 3. The trace element contents of the composite washability samples of the region averaged 0.11 ppm cadmium, 21 ppm chromium, 12 ppm copper, 58 ppm fluorine, 0.23 ppm mercury, 12 ppm manganese, 13 ppm nickel, and 6.2 ppm lead.

The washability data show that most of these trace elements concentrate in the heavier specific gravity fractions, which indicates that they are associated with the inorganic matter. Therefore, crushing the coal to 14-mesh top size and removing the sink 1.60 specific gravity material would provide significant trace element reductions ranging up to 53 percent (summary table 12).

The ratios in table 13 show that the trace element concentrations of the sink 1.60 specific gravity material were greater by factors ranging from 3 to 13, compared with those of the float 1.60 specific gravity material.

Figure 1 plots the washability data for the Pittsburgh bed coal sample collected from Pennsylvania. The curves show that generally significant trace element rejection would occur at a specific gravity of separation of about 1.40 with a clean coal recovery[1] of 88 percent.

Figure 2 plots the washability data for the Waynesburg bed coal sample collected from Ohio. The curves show that even though the cadmium and mercury contents showed a high concentration in the 1.30 specific gravity fraction, generally significant trace element reduction would occur at a specific gravity of separation of 1.60 with a clean coal recovery of 85 percent.

Figure 3 plots the washability data for the Upper Freeport bed coal sample collected from Maryland. The curves show that significant trace element reductions would occur at a specific gravity of separation of 1.60 with an attendant clean coal recovery of 85 percent. Generally, the range of the trace element contents varied considerably for the three coals tested.

[1]All recoveries are weight percent.

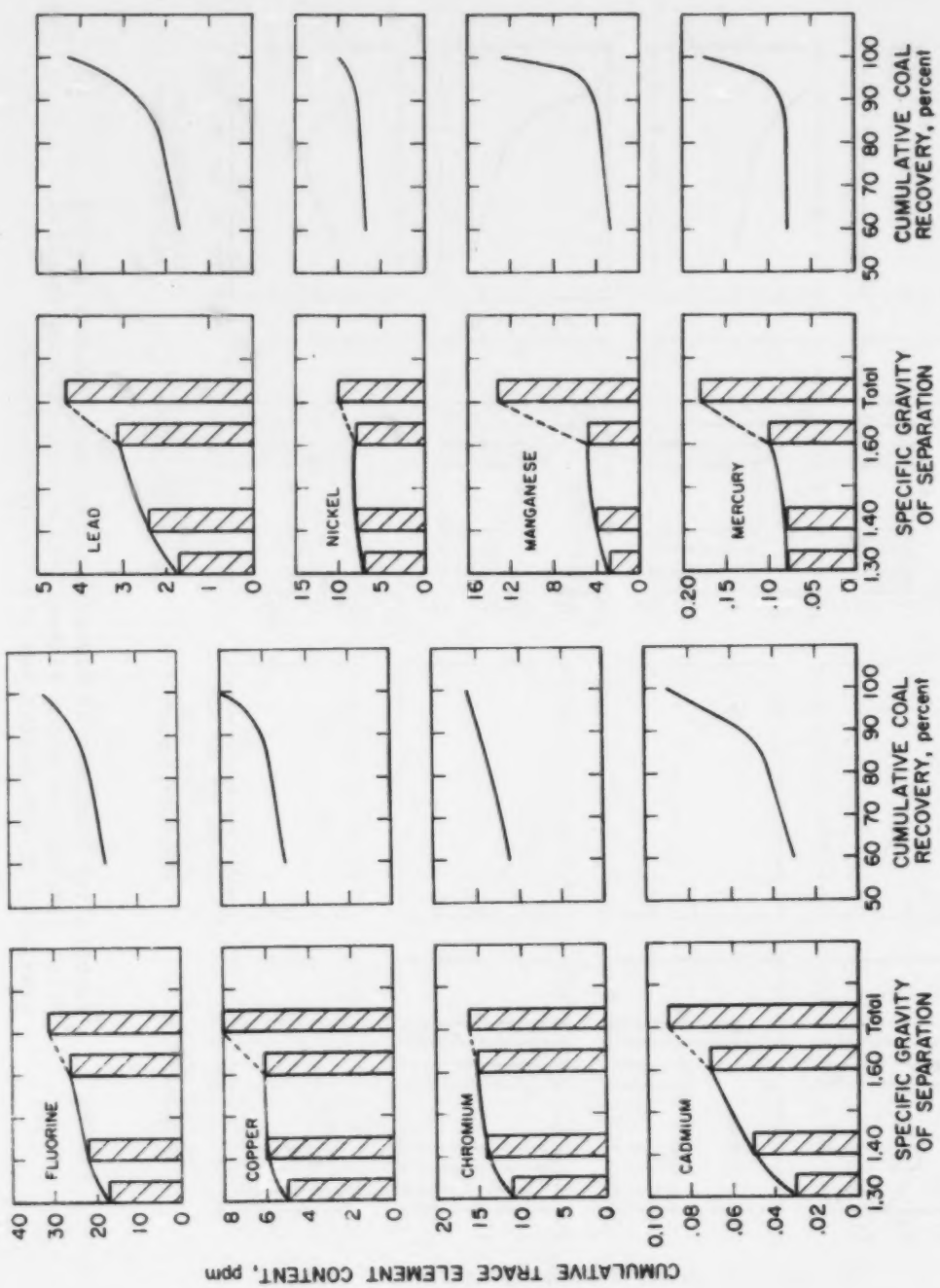

FIGURE 1. - Washability analyses of Pittsburgh bed coal, Allegheny County, Pa., showing the trace element content at various specific gravities of separation and clean coal recoveries.

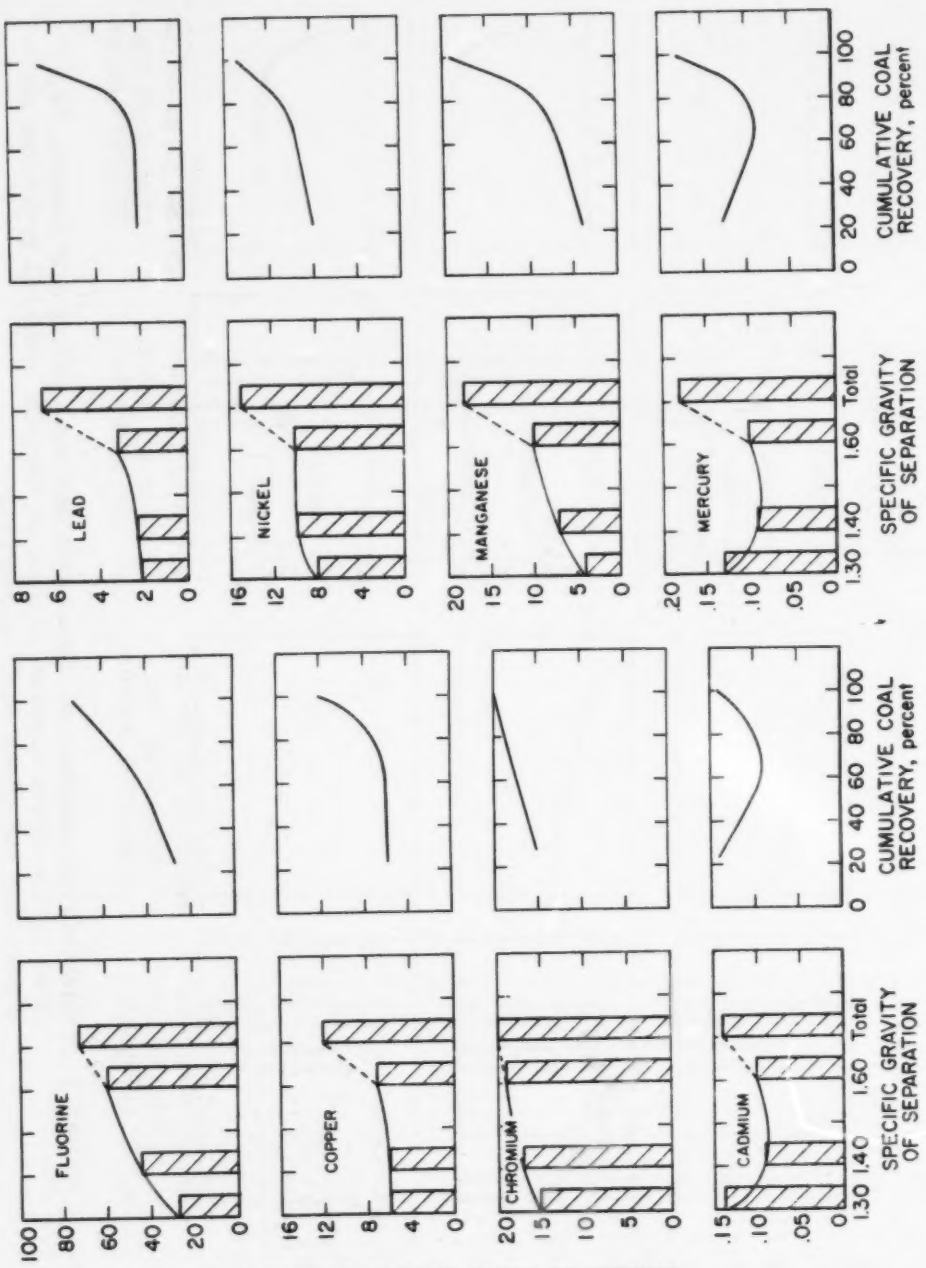

FIGURE 2. - Washability analyses of Waynesburg bed coal, Belmont County, Ohio, showing the trace element content at various specific gravities of separation and clean coal recoveries.

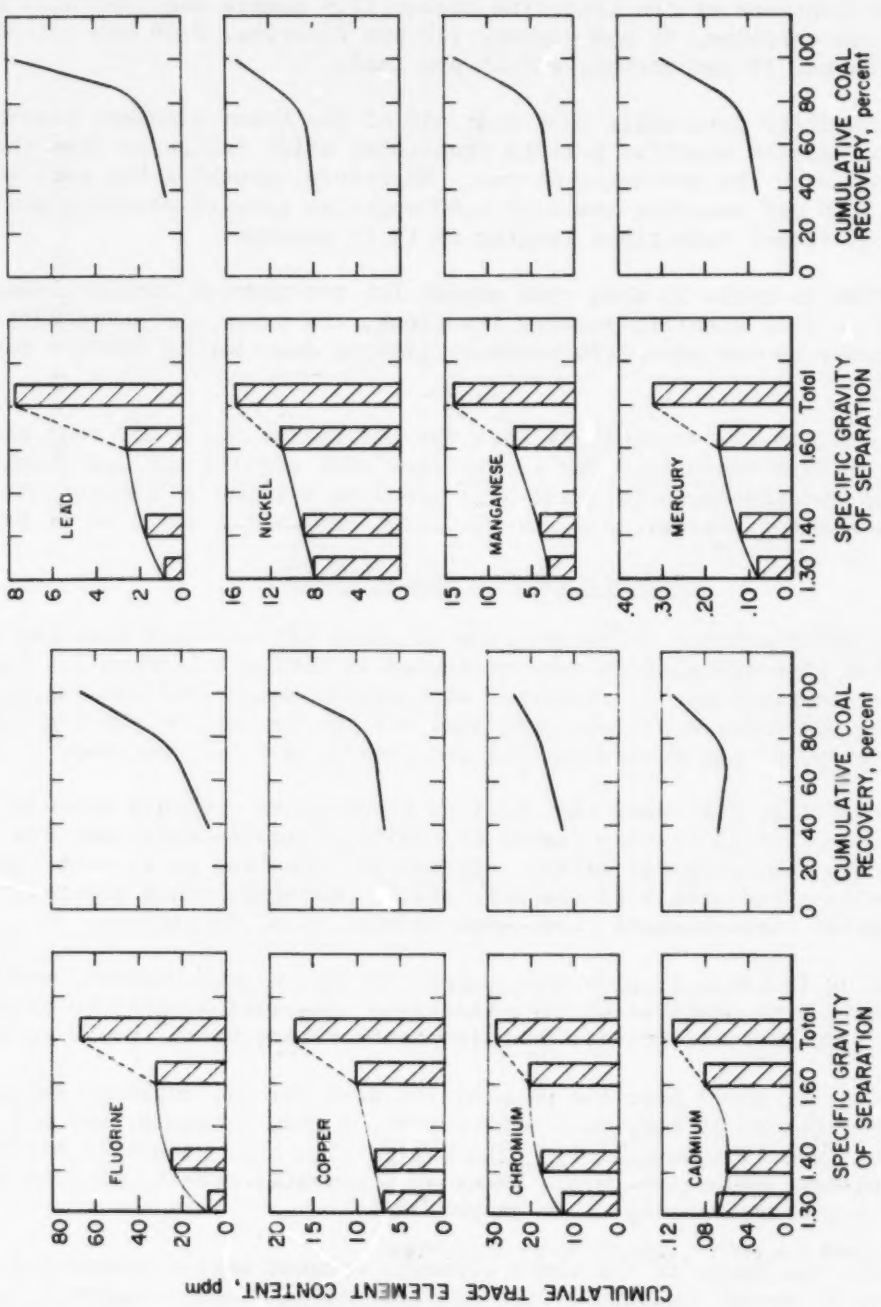

FIGURE 3. - Washability analyses of Upper Freeport bed coal, Garrett County, Md., showing the trace element content at various specific gravities of separation and clean coal recoveries.

Southern Appalachian Region Coals

A sample of Hazard No. 4 bed coal from East Kentucky was evaluated, and the washability data are presented in table 5 and plotted in figure 4. The trace element contents of the composite washability sample analyzed 0.12 ppm cadmium, 25 ppm chromium, 31 ppm copper, 112 ppm fluorine, 0.09 ppm mercury, 300 ppm manganese, 19 ppm nickel, and 15 ppm lead.

The washability data again show that all of the trace elements concentrated in the heavier specific gravity fractions, which indicates that they are associated with the inorganic matter. Therefore, crushing the coal to 14-mesh top size and removing the sink 1.60 specific gravity material would provide trace element reductions ranging up to 77 percent.

The ratios in table 13 show that except for the cadmium content, which was the same in both specific gravity fractions, the other concentrations would be greater in the sink 1.60 specific gravity fraction by factors ranging from 3 to 16.

Figure 4 plots the washability data for the Hazard No. 4 bed coal sample collected from East Kentucky. The curves show that significant and feasible trace element reductions would occur at a specific gravity of separation of 1.60 for all elements except cadmium; the clean coal yield would be 77 percent.

Eastern Midwest Region Coals

Three coalbed samples collected from Illinois (2) and West Kentucky (1) were evaluated with washability data presented in tables 6 through 8. The trace element contents of the composite washability samples of the region averaged 0.34 ppm cadmium, 12 ppm chromium, 6.3 ppm copper, 54 ppm fluorine, 0.09 ppm mercury, 47 ppm manganese, 9.8 ppm nickel, and 5.6 ppm lead.

The washability data show that most of these trace elements concentrate in the heavier specific gravity fractions, which indicates that they are associated with the inorganic matter. Therefore, crushing to 14-mesh top size and removing the sink 1.60 specific gravity material would generally provide significant trace element reductions ranging up to 88 percent.

The ratios in table 13 show that except for the nickel content, which was the same in both specific gravity fractions, the ratios would be greater in the sink 1.60 specific gravity fraction by factors ranging from 2 to 320.

Figures 5, 6, and 7 plot the washability data for the coalbed samples collected from the No. 6 bed, Illinois, the No. 5 bed, Illinois, and the No. 7 bed, Kentucky (West), respectively. The curves show that generally significant trace element reductions would occur at a specific gravity of 1.60 with clean coal recoveries ranging up to 94 percent.

Generally the range of the trace element contents varied considerably for the three coals tested, especially the cadmium content, which ranged from 0.04 to 0.64 ppm, and the manganese content, which ranged from 13 to 88 ppm.

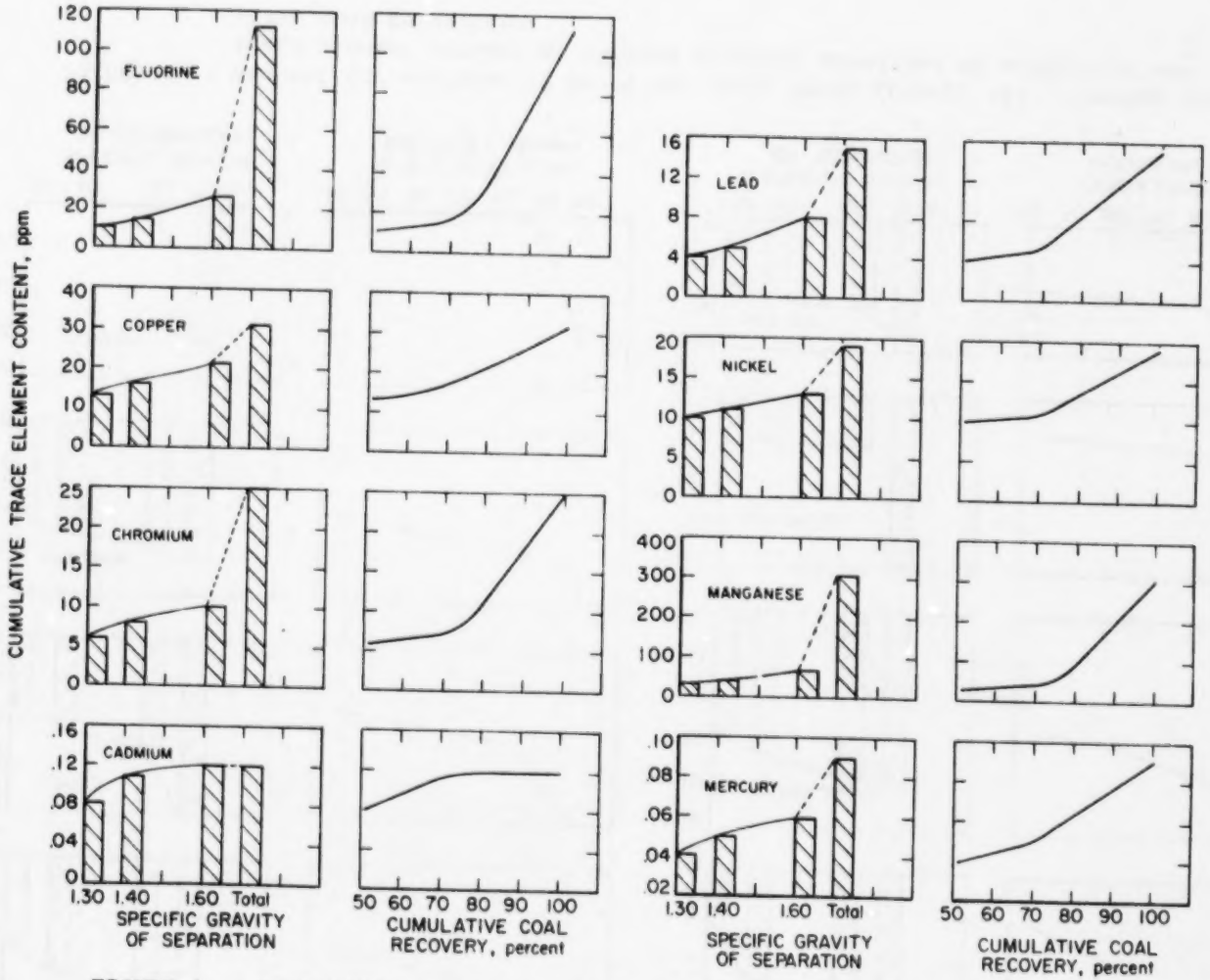

FIGURE 4. - Washability analyses of Hazard No. 4 bed coal, Bell County, Ky. (East), showing the trace element content at various specific gravities of separation and clean coal recoveries.

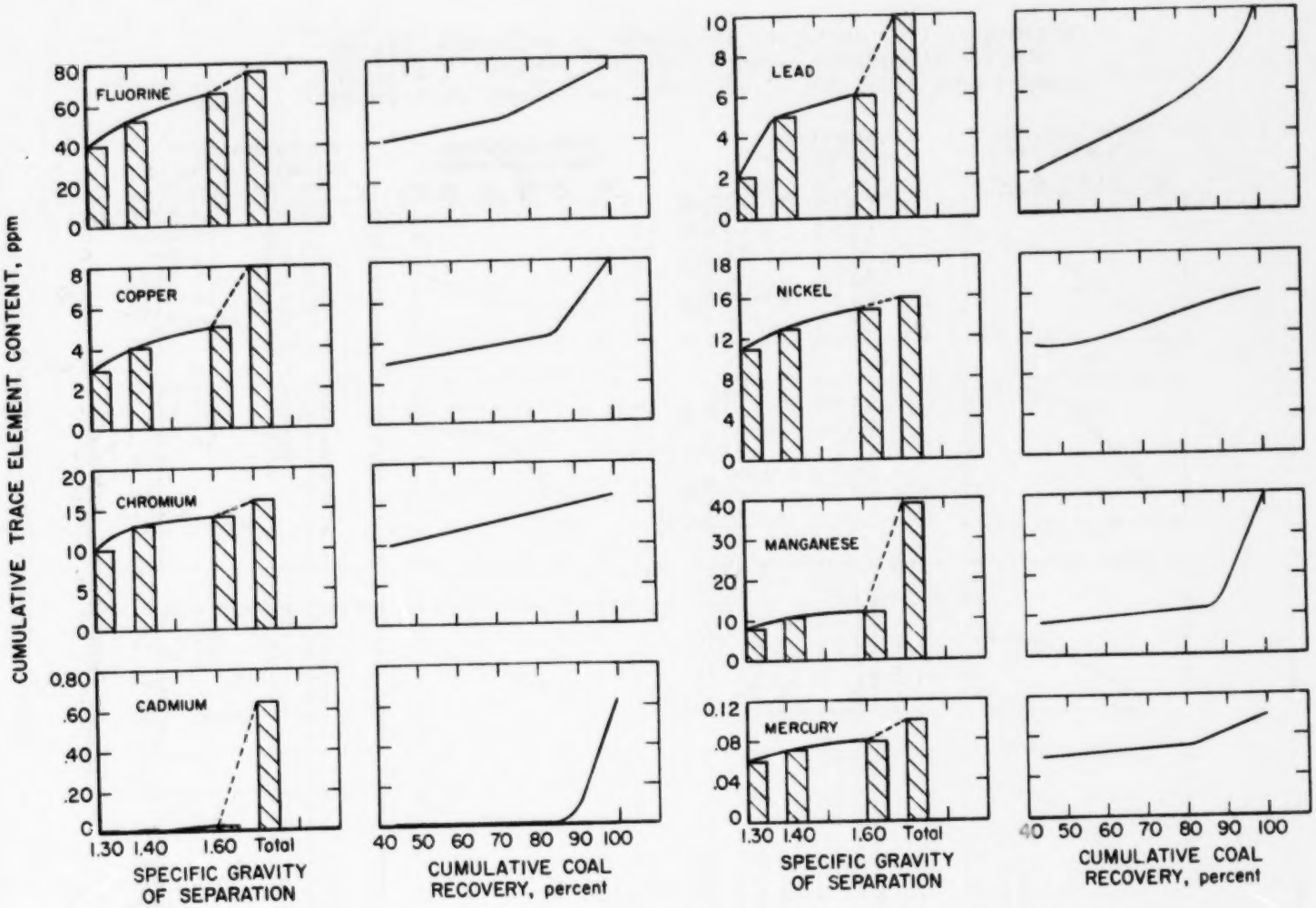

FIGURE 5. - Washability analyses of No. 6 bed coal, Perry County, Ill., showing the trace element content at various specific gravities of separation and clean coal recoveries.

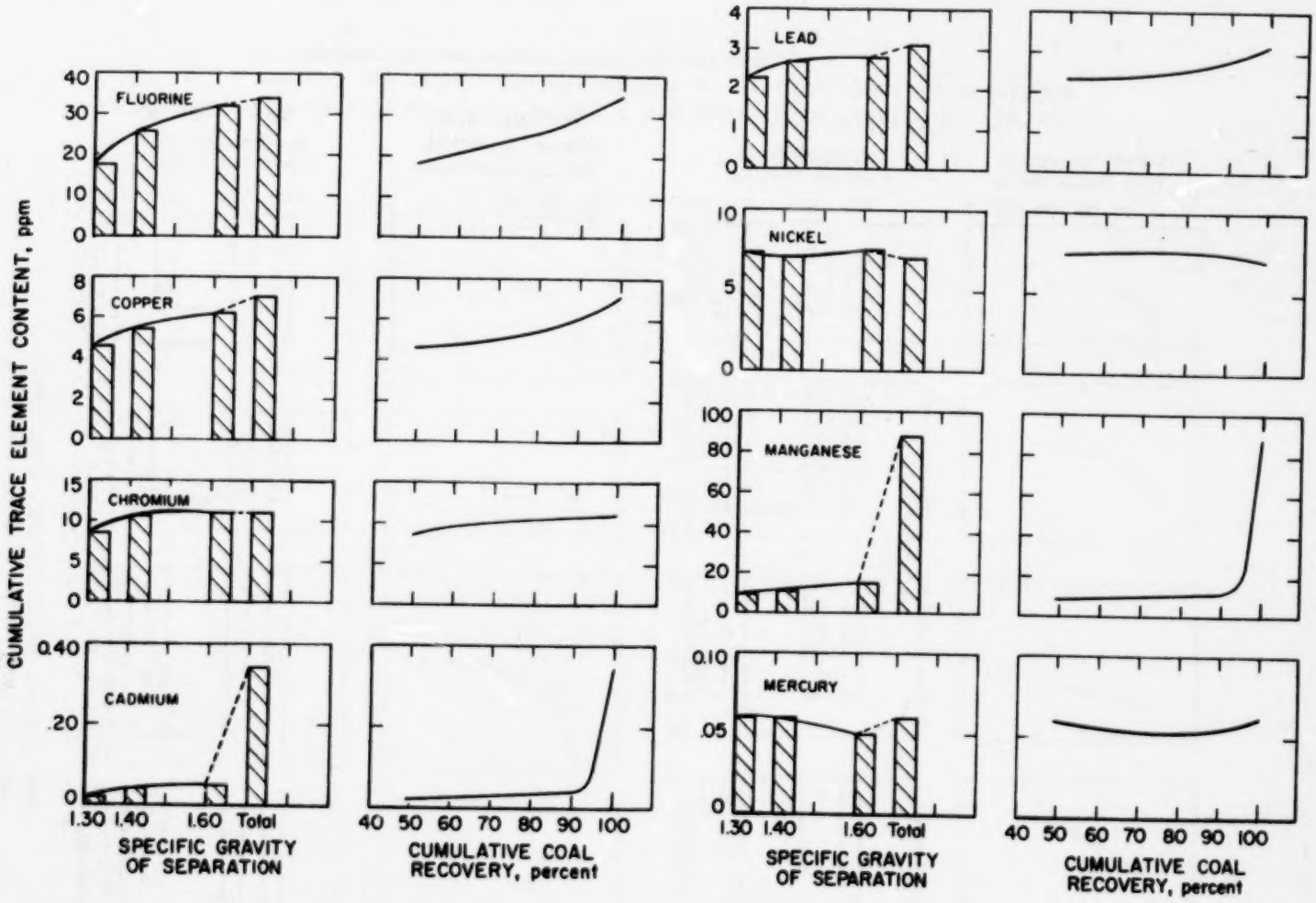

FIGURE 6. - Washability analyses of No. 5 bed coal, Perry County, Ill., showing the trace element content at various specific gravities of separation and clean coal recoveries.

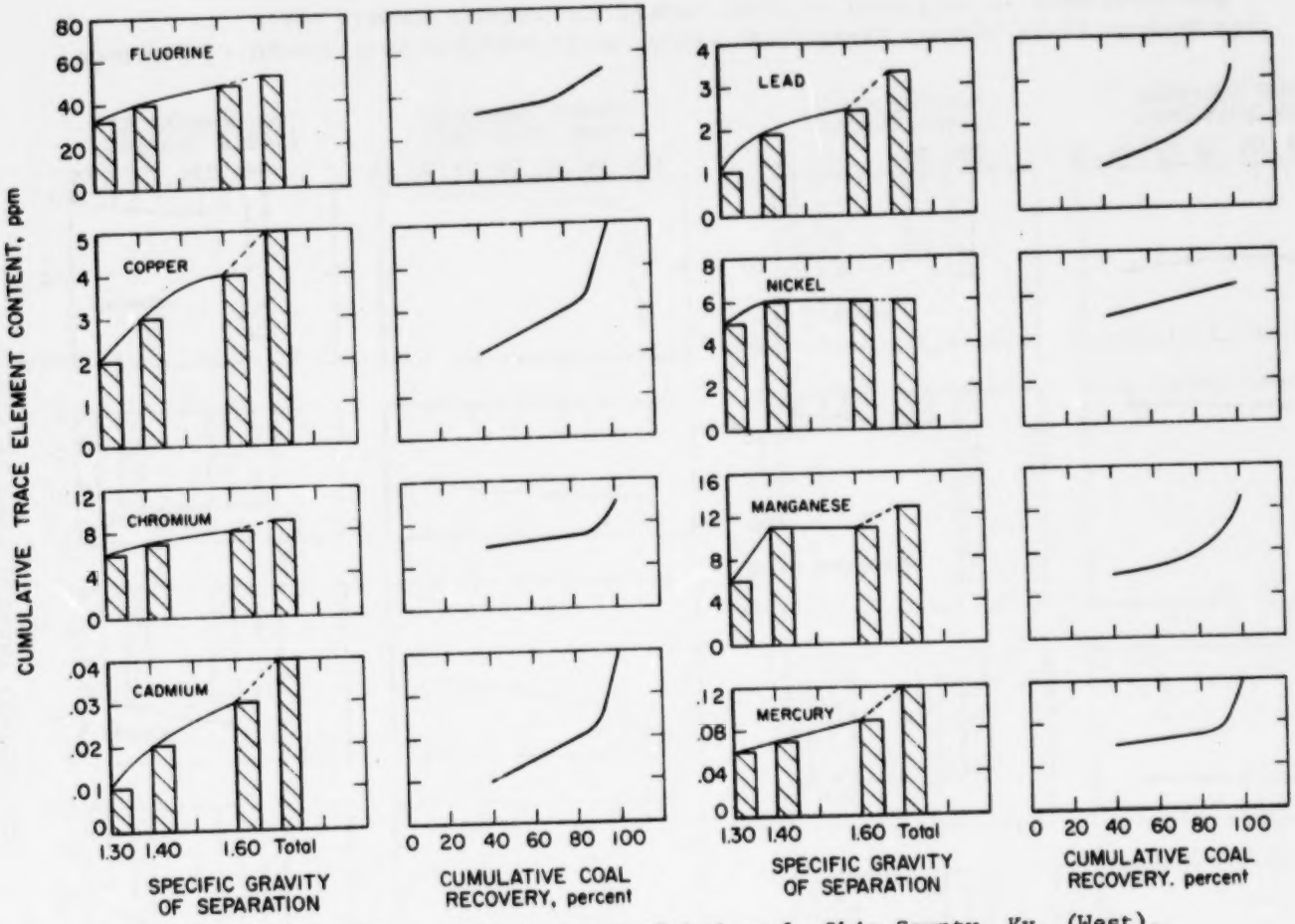

FIGURE 7. - Washability analyses of No. 7 bed coal, Ohio County, Ky. (West), showing the trace element content at various specific gravities of separation and clean coal recoveries.

Western Region Coals

Three coalbed samples collected from Arizona (1), New Mexico (1), and Wyoming (1) were evaluated with washability data presented in tables 9 through 11 and plotted in figures 8 through 10. The trace element contents of the composite washability samples of the region averaged 0.10 ppm cadmium, 3.7 ppm chromium, 7.6 ppm copper, 40 ppm fluorine, 0.06 ppm mercury, 45 ppm manganese, 4.1 ppm nickel, and 7.1 ppm lead.

The composite washability data show that most of the trace elements concentrated in the heavier specific gravity fractions. Therefore, removing the sink 1.60 specific gravity material would provide significant trace element reductions ranging up to 64 percent.

Table 13 shows that the trace element concentrations were greater in the sink 1.60 specific gravity fraction by factors ranging from 2 to 19.

Figures 8, 9, and 10 plot the washability data for the coalbed samples collected from the Red bed, Arizona, the No. 8 bed, New Mexico, and the Rock Springs No. 3 bed, Wyoming. The curves show that generally significant trace element reductions would occur at a specific gravity of separation of 1.40 at clean coal recoveries ranging up to 89 percent for the coals from Arizona and Wyoming, compared with 1.60 specific gravity of separation with a clean coal recovery of 82 percent for the coal from New Mexico. Generally the three coals of this region also showed a wide range in the levels of trace element content.

The ratios presented in table 13 show that the manganese had the greatest concentration in the sink 1.60 specific gravity fraction of the coals for the four regions tested, by factors ranging from 13 to 35. An exception to this was the cadmium concentration in the sink 1.60 specific gravity fraction of the Eastern Midwest region coals, which was greater by a factor of 310.

CONCLUSIONS

1. Reliable analytical techniques were developed to determine cadmium, chromium, copper, fluorine, mercury, manganese, nickel, and lead contents in the whole coal as well as the various specific gravity fractions of the coal. The bias of the results produced by the developed techniques ranged from 0 to 17 percent for the various trace elements when comparing the determined values with those certified by the National Bureau of Standards for SRM 1632. The precision of the developed techniques was ±15 percent or less when comparing the cumulative trace element contents of the various specific gravity fractions of a coal with those obtained from the whole coal.

2. Contamination of samples can occur from lead in automobile exhaust products, mercury vapor in tanks of laboratory gases, and laboratory equipment such as beakers and stirring rods.

3. The method of standard additions was found most acceptable for determining the trace element content of the various specific gravity fractions of the coals tested.

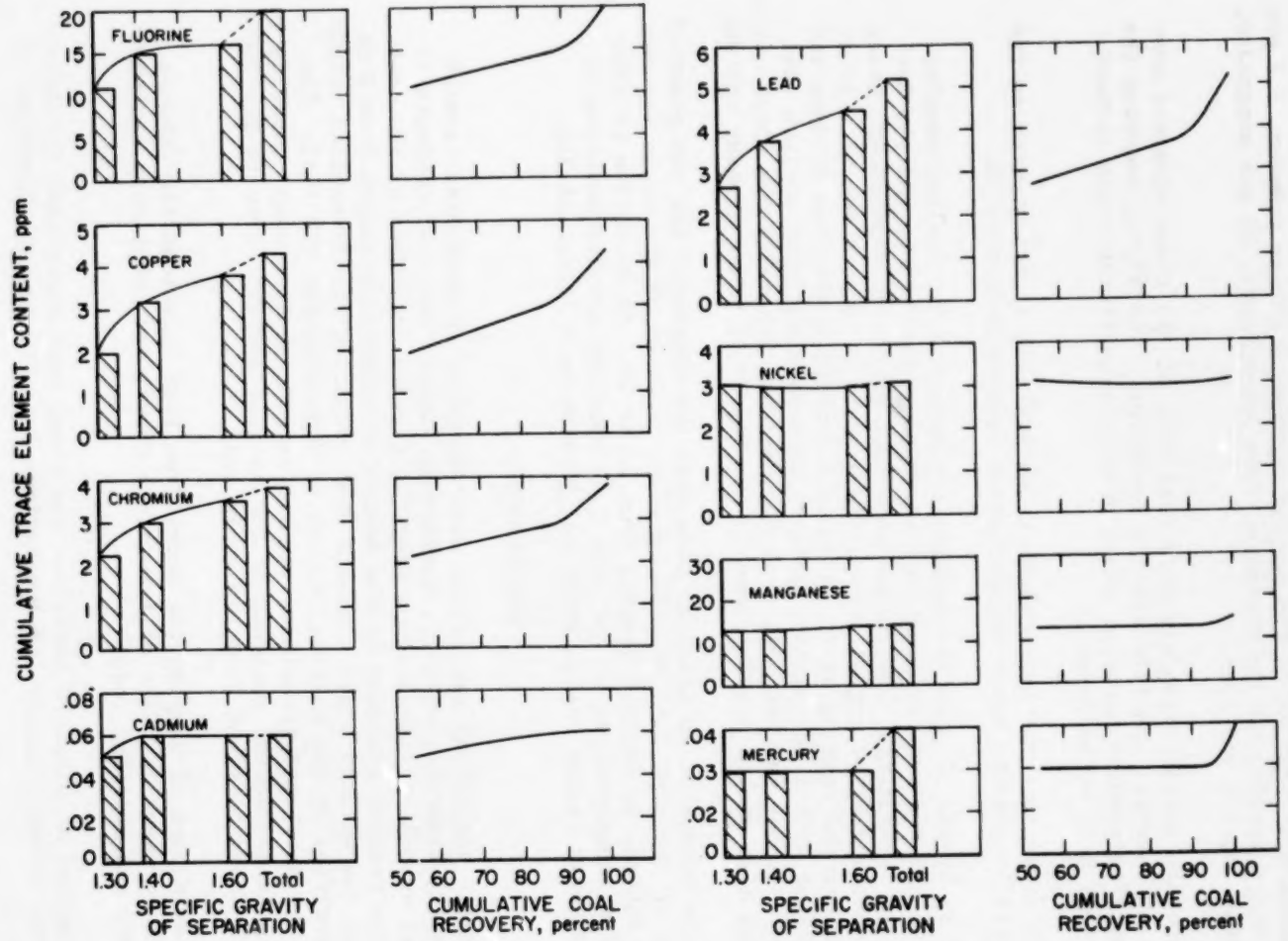

FIGURE 8. - Washability analyses of Red bed coal, Navajo County, Ariz., showing the trace element content at various specific gravities of separation and clean coal recoveries.

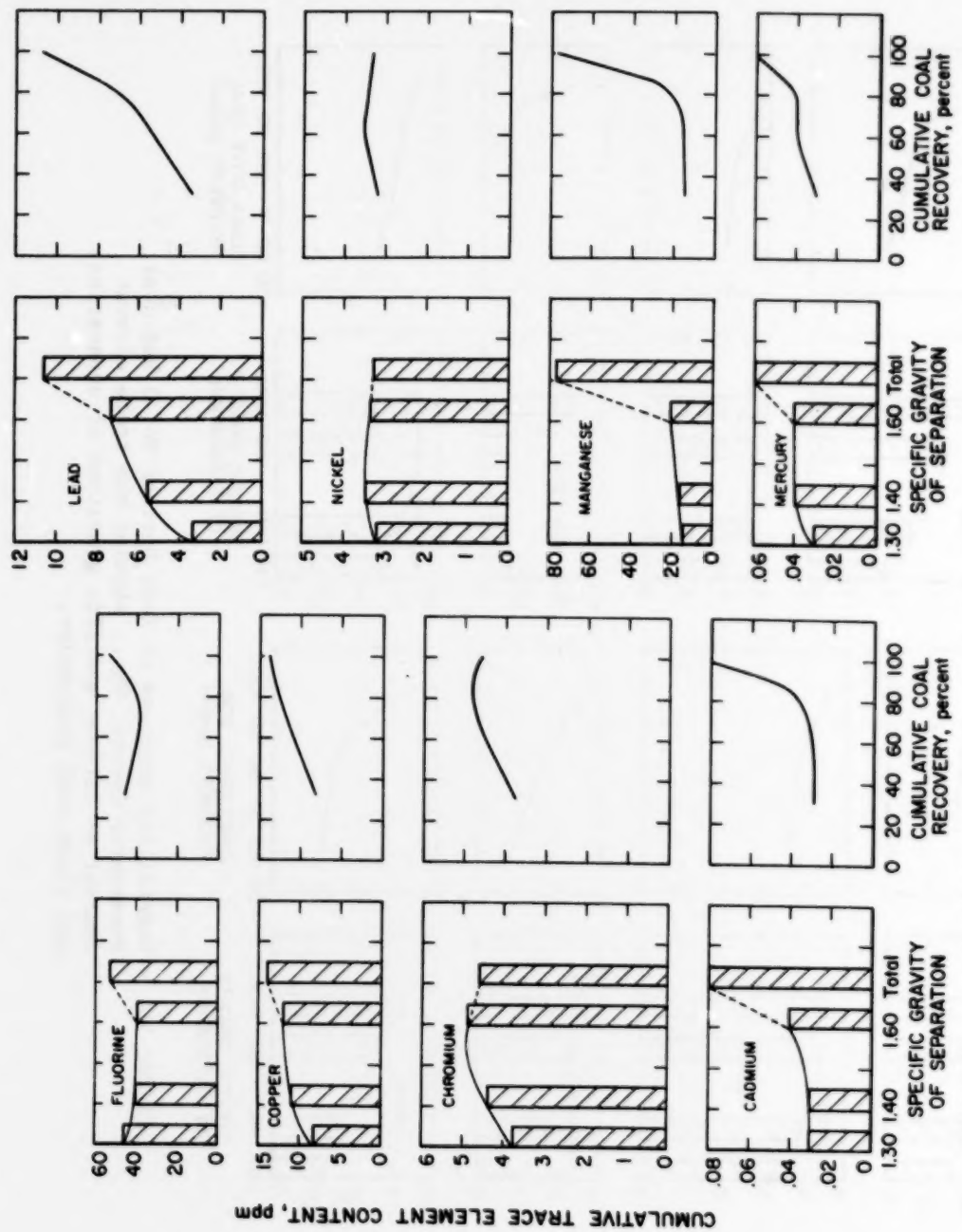

FIGURE 9. - Washability analyses of No. 8 bed coal, San Juan County, N. Mex., showing the trace element content at various specific gravities of separation and clean coal recoveries.

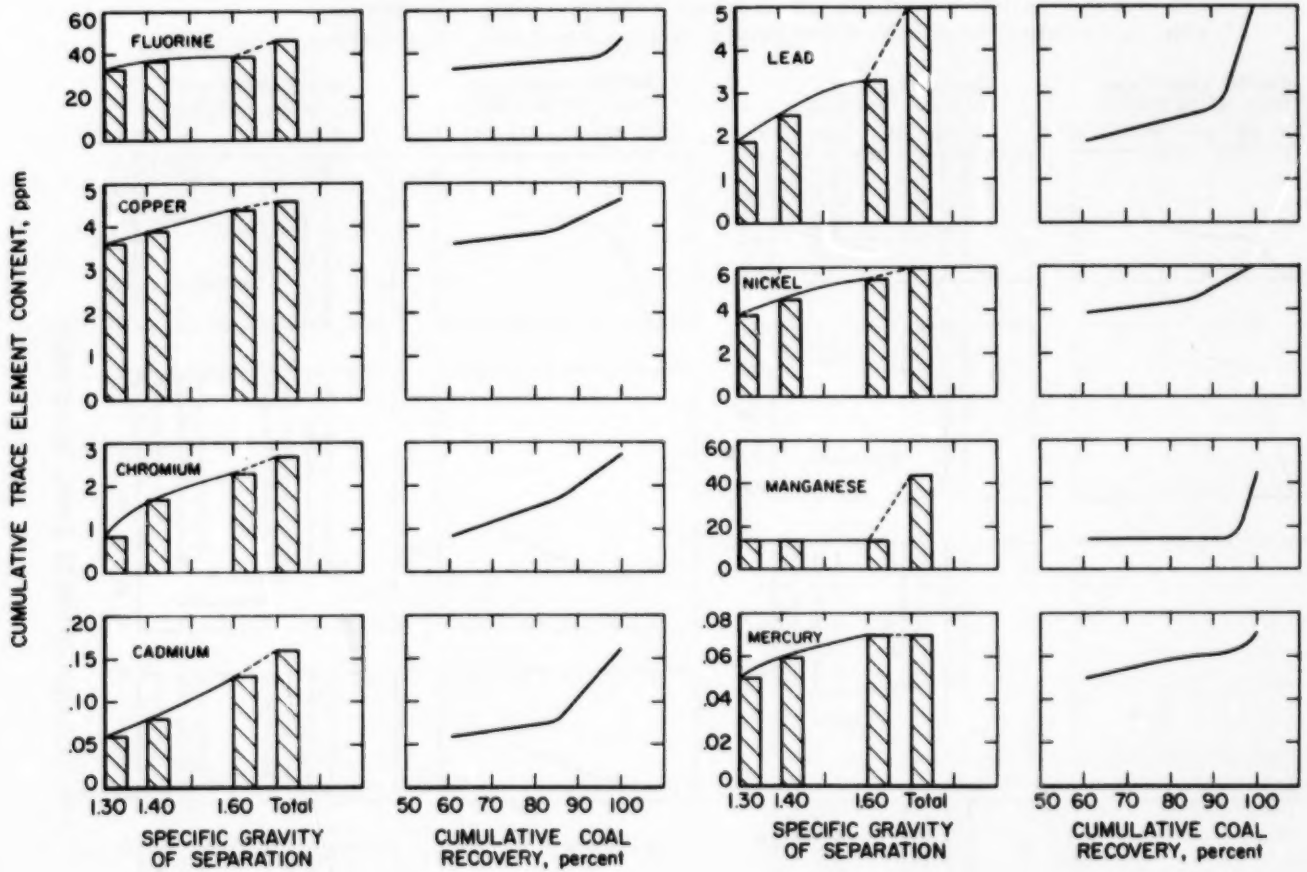

FIGURE 10. - Washability analyses of Rock Springs No. 3 bed coal, Sweetwater County, Wyo., showing the trace element content at various specific gravities of separation and clean coal recoveries.

4. Washability analyses performed on the coals showed that most of the trace elements presented in this report concentrated in the heavier specific gravity fractions of the coal, indicating that they are associated with the inorganic matter. Thus removal of these heavier gravity fractions would result in significant trace element reductions in the clean coal product.

5. The concentrations of the individual trace elements varied quite a bit for the various coalbeds within a region and thus for the various regions also. However, in most instances the concentration ratios for cadmium, chromium, copper, fluorine, mercury, nickel, and lead were 1/10 or less, while that of manganese was always greater than 1/10 for the coals tested in all four regions.

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16. ABSTRACT The report gives results of a washability study showing the trace element contents of various specific gravity fractions for ten coal samples collected from four coal producing regions of the U.S. Reliable analytical methods were developed to quantitatively determine cadmium, chromium, copper, fluorine, mercury, manganese, nickel, and lead in whole coals. Generally, the data showed that most of the trace elements of interest concentrated in the heavier specific gravity fractions of the coal, indicating that they are associated with mineral matter. Removing this material should reduce trace elements significantly.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	Pollution Control	13B
Coal	Stationary Sources	08G
Trace Elements	Washability	06A
Chemical Analysis		07D
Analyzing		14B
Washing		13H, 07A

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